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## PATENT ABSTRACTS OF JAPAN

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# (54) COATING MATERIAL AND METHOD OF FORMING COATING FILM USING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a coating material affording the surface of an object member with high abrasion resistance and smoothness when applied on the surface.

SOLUTION: The coating material is obtained by blending at least one fluororesin selected from a tetrafluoroethylene-perfluoroalkyl vinyl ether copolymer resin(PFA), an ethylene-tetrafluoroethylene copolymer resin(ETFE) and a tetrafluoroethylene-hexafluoropropylene copolymer resin(FEP) or a

polyamide-imide as a coating resin with 5-50 wt.% of a modified fluororesin obtained by irradiating an FEP heated to 80-280°C lower than the melting point of the

FEP with radiation under the conditions of ≤13 kPa in

oxygen concentration and 1 kGy to 10 MGy in absorption dose. A coating film is formed by coating an object member with this coating material followed by baking the coating material.



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#### DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

This invention relates to the formation method of the coating film using the charge of a finishing material and the charge of a finishing material which are used for surface coating of a forming mold, etc.

[0002]

[Description of the Prior Art]

In recent years, the smooth coating film excellent in abrasion resistance is called for. For example, surface coating of the forming mold is asked for the smooth coating film excellent in abrasion resistance. This is because the abrasion resistance of a coating film is required in order to heat and slush the material of a cast into a metallic mold at the time of shaping, and the smooth nature of a coating film is called for in order to lessen unevenness on the surface of mold goods.

[0003]

In the charge of a finishing material, conventionally Tetrafluoroethylene perfluoroalkyl vinyl ether copolymerization resin (PFA), At least one fluororesin chosen from ethylene-tetrafluoroethylene copolymerization resin (ETFE) and tetrafluoroethylene hexafluoropropylene copolymerization resin (FEP) and resin for paint by polyamidoimide have been used. However, although smooth nature is good, since abrasion resistance was inferior in the resin for paint, there was a fault that a life when it paints to a sliding part was short.

[0004]

At least one mother agent fluororesin chosen from PFA, ETFE, and FEP in order to cancel this fault, By painting and calcinating a mixture with the refining fluororesin produced by irradiating with an ionizing radiation the unmodified fluororesin heated more than the melting point, the

mold-release characteristic of a painted surface is good, and there is the method of lengthening a life by what abrasion loss is lessened for (abrasion resistance is raised). [0005]

[Problem(s) to be Solved by the Invention]

However, in the formation method of the coating film using the conventional charge of a finishing material mentioned above, although the abrasion resistance of the painted surface improved, there was a problem that surface roughness was large. For this reason, for example, in using as a surface-coating material for forming molds, it is necessary to make a painted surface smooth, and metallic mold production expense becomes high by polish. [0006]

This invention is made in view of this point, and is a thing.

The purpose is to provide the formation method of the coating film using the charge of a finishing material and the charge of a finishing material which can obtain the abrasion resistance which was excellent when it painted on the surface of \*\*, and smooth nature.

### [0007]

[Means for Solving the Problem]

In order to solve an aforementioned problem, a charge of a finishing material of this invention is characterized by mixing resin for paint, and a refining fluororesin which reformed tetrafluoroethylene hexafluoropropylene copolymerization resin (FEP) at 5 to 50% of the weight of a rate.

[8000]

Said resin for paint Tetrafluoroethylene perfluoroalkyl vinyl ether copolymerization resin (PFA), It is characterized by being at least one fluororesin chosen from ethylene-tetrafluoroethylene copolymerization resin (ETFE) and tetrafluoroethylene hexafluoropropylene copolymerization resin (FEP), or polyamidoimide.

[0009]

Said refining fluororesins are the conditions of 13 or less kPa of oxygen densities, absorbed dose 1kGy - 10MGy, and are characterized by being obtained by irradiating with radiation FEP heated by 80-280 \*\* below the melting point.

[0010]

A formation method of a coating film using a charge of a finishing material of this invention, On condition of at least one fluororesin chosen from PFA, ETFE, and FEP or resin for paint which is polyamidoimide, 13 or less kPa of oxygen densities and absorbed dose 1kGy - 10MGy, It is characterized by calcinating it, after painting a charge of a finishing material which mixed a refining fluororesin produced by irradiating with radiation FEP heated by 80-280 \*\* below the melting point at 5 to 50% of the weight of a rate to a member to be painted.

[0011]

[Embodiment of the Invention]

Hereafter, an embodiment of the invention is described in detail with reference to drawings. [0012]

(Embodiment)

<u>Drawing 1</u> is a figure showing an example of the thin film which exfoliated after applying monotonously the charge of a finishing material concerning an embodiment of the invention. <u>Drawing 2</u> is a figure showing an example of the thin film which exfoliated after applying the charge of a finishing material concerning an embodiment of the invention to the inside of a cylinder.

[0013]

The charge of a finishing material of this embodiment applied for forming the cylindrical thin film (coating film) 20 shown in the plate-like thin film (coating film) 10 or <u>drawing 2</u> shown in <u>drawing 1</u> is the mixture which contained the resin for paint mentioned later, and a refining fluororesin at 5 to 50% of the weight of a rate. A thin film (coating film) is obtained by painting and calcinating the charge of this finishing material to a plate or a cylindrical member to be painted.

[0014]

Namely, at least one fluororesin in which the charge of this finishing material was chosen from PFA, ETFE, and FEP, Or the refining fluorine resin powder produced by the powder, the solution, or dispersion liquid of polyamidoimide by irradiating with radiation FEP heated by 80-280 \*\* on condition of 13 or less kPa of oxygen densities, absorbed dose 1kGy - 10MGy can be blended and obtained.

[0015]

As the radiation, the radiation to which crosslinking reaction (reaction which \*\*\*\*\*\* between chains, such as polymers, by a chemical bond) of the unmodified fluoro-resin can be carried out, for example, an ionizing radiation with an ionizing action, is preferred. Specifically, a gamma ray, an electron beam, X-rays, a neutron beam, or high energy ion is mentioned. [0016]

It is preferred to face to irradiate with an ionizing radiation and to heat an unmodified fluororesin below to the crystalline melting point. That is, it is preferred to irradiate with an ionizing radiation a temperature lower than 275 \*\* which is a crystalline melting point of FEP, where PTFE is heated. If an unmodified fluororesin is heated more than the melting point, it is difficult for resin to weld and to make it 50 micrometers or less of maximum droplet sizes with the mean particle diameter of 10 micrometers or less by grinding. Since crosslinking reaction occurs by heating below to a crystalline melting point unlike other fluororesins, and irradiating with an ionizing radiation, grinding of 50 micrometers or less of maximum droplet sizes of FEP

is attained with the mean particle diameter of 10 micrometers or less. [0017]

As for the exposure conditions of an ionizing radiation, 13 or less kPa of oxygen densities are conditions of absorbed dose 1kGy of an unmodified fluoro-resin - 10MGy under the inert gas atmosphere of 1.3 or less kPa preferably. If oxygen density 13kPa is exceeded, the radical etc. which were produced by radiation irradiation will be consumed and crosslinking reaction will not progress. It becomes it difficult to produce crosslinking reaction to be 1 or less kGy of absorbed doses, if 10MGy is surpassed, the remarkable fall of elongation etc. will be imitated, and it is \*\*.

[0018]

The solution or dispersion liquid of resin for paint in which the above-mentioned refining fluororesin powder is blended is obtained by at least one fluororesin chosen from PFA, ETFE, and FEP or polyamidoimide being independent, dissolving, or making a solution distribute a mixture.

[0019]

The example and comparative example at the time of actually manufacturing the charge of a finishing material of such this embodiment are explained.

[0020]

To an aluminum board, PFA (die KINE business incorporated company make; ACX-31, particle diameter of 25 micrometers, melting point of 305 \*\*), The resin whole quantity is received in the refining fluorine resin powder (Hitachi Cable, Ltd. make; the pulverization XFO, 20 micrometers of maximum droplet sizes) of this embodiment, What 0-70 capacity % combination made respectively with 100 - 30 capacity % was sprayed, it calcinated for 50 minutes at 360 \*\*, and the coating film of Examples 1-5 and the comparative examples 1-3 which are shown in the following table 1 was obtained.

[0021]

It is ETFE (die KINE business incorporated company make; EC-6520 and the particle diameter of 50 micrometers) about a compounding agent. The melting point of 225 \*\*, and FEP (die KINE business incorporated company make; NCX-1 and the particle diameter of 25 micrometers) The coating film of Examples 6-15 and the comparative examples 4-9 which are changed into the melting point of 250 \*\* and PAI (Hitachi Chemical [ Co., Ltd. ] make: HPC-6000A, viscosity of 1300 mPa-s/25 \*\*), and are similarly shown in the following table 2 was obtained. The thickness of the coating film was 40-80 micrometers.

After measuring the surface roughness of the obtained coating film, it hit against the ring surface made from SUS304 which ground surface roughness to Ra0.2micrometer, and the ring one disk examination estimated relative abrasion loss. 10.8 m/min and measurement

temperature are 25 \*\*, and load is [ 980N and the measuring time of a test condition of speed ] 30 minutes. A measurement result is shown in the tables 1 and 2. [0023]

[Table 1]

大数の		ſì⊈	70	0	30	0.1	-
比較例 4		ባኞ ባኞ	02	0	30	1.1	1.4
比較例 3			100	0	0	0.4	10
実 <b>施例</b> 5			95	5	0	0.4	1.2
実施例 4	揪		06	10	0	0.4	1.1
実施例 3	静電粉体		80	20	0	0.4	1.1
実施例 2			09	40	0	0.4	-
実施例 1			09	20	0	0.4	į
比較例 2			40	09	0	0.4	3
比較例 1			90	02	0	0.4	10
	<b>拳</b> 布方法	中	PFA	微粉化XFO	XF0	表面粗さ(μm)	相対摩耗量

[Table 2]

<b>€</b>	ン業								
比較 14	スプレ   静電粉体塗装   <sup>スプ</sup>  -塗装		0	0	30	0	70	1.3	വ
比較例 13			0	30	0	0	20	1.4	2
比較例 12			30	0	0	0	70	1.2	33
比較例 11			0	0	100	0	0	6.0	20
比較例 10	静電粉体塗装 スプレ   静電粉体塗装   スプレ   静電粉体塗装		0	100	0	0	0	0.4	0
比較例 9		Ú\$	100	0	0	0	0	8.0	15
比較例 8			0	0	30	02	0	0.3	21
比較例 7			0	30	0	20	0	0.4	8
比較例 6			30	0	0	02	0	8.0	- 21
実施例 8			0	0	70	30	0	0.3	5
実施例 7			0	02	0	90	0	0.4	7
実施例 6			70	0	0	30	0	0.8	2
	塗布方法	中層	ETFE	FEP	PAI	微粉化XFO	XF0	<b>長面粗さ(μm)</b>	相対摩耗量

The examining result at the time of setting resin for paint to PFA was shown in Table 1. As shown in Example 5 from Example 1, as for the blending ratio of the pulverization XFO, in 5 - 50 capacity %, surface roughness and relative abrasion loss become good to the whole coat. if the blending ratio of the pulverization XFO surpasses 50 capacity % as shown in the

comparative example 1 and the comparative example 2 -- the surface -- a uniform coat is hard to be obtained and relative abrasion loss becomes large. As shown in the comparative example 3, abrasion resistance and endurance do not improve that the blending ratio of the pulverization XFO is less than 5 capacity %. As shown in the comparative example 4 and the comparative example 5, when XFO is blended, relative abrasion loss is the same as the case where the pulverization XFO is blended, but surface roughness becomes large without polish. [0024]

The examining result at the time of setting resin for paint to ETFE, FEP, and PAI was shown in Table 2. As shown in Example 8 from Example 6, surface roughness and relative abrasion loss become good by blending the pulverization XFO. 5 - 50 capacity % of a blending ratio is preferred to the whole coat. if the blending ratio of the pulverization XFO exceeds 50 capacity % as shown in the comparative example 8 from the comparative example 6 -- the surface -- a uniform coat is hard to be obtained and relative abrasion loss becomes large. [0025]

As shown in the comparative example 11 from the comparative example 9, abrasion resistance and endurance do not improve that the blending ratio of the pulverization XFO is less than 5 capacity %. As shown in the comparative example 14 from the comparative example 12, when XFO is blended, relative abrasion loss is the same as the case where the pulverization XFO is blended, but surface roughness becomes large without polish. [0026]

Thus, according to the formation method of the coating film using the charge of a finishing material and the charge of a finishing material of this embodiment. First, on condition of at least one fluororesin chosen from PFA, ETFE, and FEP or resin for paint which is polyamidoimide, 13 or less kPa of oxygen densities and absorbed dose 1kGy - 10MGy, The charge of a finishing material was obtained by mixing the refining fluororesin produced by irradiating with radiation FEP heated by 80-280 \*\* below the melting point at 5 to 50% of the weight of a rate.

[0027]

Thus, since the refining fluororesin contained is the refining fluorine resin powder of 50 micrometers or less of maximum droplet sizes in the mean particle diameter of 10 micrometers or less, the obtained charge of a finishing material becomes the thing excellent in the dispersibility to a coating film, or adhesion with a resin paint film.

[0028]

Therefore, the coating film obtained by calcinating it after painting this charge of a finishing material to a member to be painted has high abrasion resistance, and it can use surface roughness of a coating film as resin for paint below equivalent, without grinding. For example, since smoothing of the painted surface by polish like before is unnecessary when it uses as a

surface-coating material for forming molds, metallic mold production expense can be made cheap.

[0029]

[Effect of the Invention]

At least one fluororesin which is chosen from PFA, ETFE, and FEP according to this invention as explained above, or resin for paint which is polyamidoimide, The charge of a finishing material which mixed the refining fluororesin produced by irradiating with radiation FEP heated by 80-280 \*\* below the melting point on condition of 13 or less kPa of oxygen densities, absorbed dose 1kGy - 10MGy at 5 to 50% of the weight of a rate was obtained. Since the refining fluororesin contained is the refining fluorine resin powder of 50 micrometers or less of maximum droplet sizes in the mean particle diameter of 10 micrometers or less, this charge of a finishing material becomes the thing excellent in the dispersibility to a coating film, or adhesion with a resin paint film. Therefore, if a coating film is formed by calcinating it after painting this charge of a finishing material to a member to be painted, it will become a coating film which has the outstanding abrasion resistance and smooth nature.

[Brief Description of the Drawings]

[Drawing 1]It is a figure showing an example of the thin film which exfoliated after applying monotonously the charge of a finishing material concerning an embodiment of the invention. [Drawing 2]It is a figure showing an example of the thin film which exfoliated after applying the charge of a finishing material concerning an embodiment of the invention to the inside of a cylinder.

[Description of Notations]

10 A plate-like thin film (coating film)

20 A cylindrical thin film (coating film)

[Translation done.]